DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention]This invention relates to the low-temperature process of an alumina crystalline thin film.
Abrasion proof [in / machinery industry, semiconductor industry, etc. / in more detail], the alumina hard coating aiming at protection, Or it is related with the low-temperature formation art of the alumina crystalline thin film for providing the cheap alumina substrate of the quality of high, and the characteristic is most excellent especially and it is related with the low-temperature formation art of alpha phase alumina membrane with sufficient stability.

[0002]
[Description of the Prior Art]Conventionally, since it was necessary to make a substrate into an about 1000 ** elevated temperature, the kind of base material by which a tunic is carried out was usually greatly restricted to formation of alpha phase alumina membrane by a CVD method (the chemicals steamy depositing method). In order to lower the forming temperature of alumina membrane in recent years, some PVD (the physical steamy depositing method) is developed, but also in alpha phase alumina membrane formation by the suitable pulse direct-current magnetron sputtering method for industrialization, the substrate temperature of at least 760 ** is [even if] indispensable. [1O. Zywitzky, G.Hoetsch, F.Fietzke, andK.Goedicke:Surface and Coatings Technology, Vol.82, 169 (1995)] .Although formation of the alumina crystalline thin film by the ionization magnetron sputtering method developed for the purpose of low-temperature formation is reported for substrate temperature to have fallen to 500 ** as a method similar to this, the crystal phase of a formation thing is kappa phase alumina which is not alpha phase but a metastable phase. [2J.Schneider, W.Sproul, A.Voevodin, and A.Matthews:J.Vac.Sci.Technol., A15, 1 (1997)] .As mentioned above, since the elevated temperature of 760 ** was conventionally needed for formation of alpha phase alumina membrane by a CVD method at 1000 ** and alpha phase alumina membrane formation by a PVD sputtering technique, the application range of the alumina crystalline thin film was restricted greatly.
[0003]
[Problem(s) to be Solved by the Invention]this invention person aims at developing the low-temperature process of alumina membrane in such a situation, As a result of repeating research wholeheartedly using the suitable sputtering technique for industrial production, by forming a chrome oxide crystalline thin film in a substrate or the base material by which a tunic is carried out beforehand, and forming an alumina crystalline thin film on it, it finds out that the desired end can be attained and came to complete this invention. That is, an object of this invention is to provide the new low-temperature process by the sputtering technique of an alumina crystalline thin film.
[0004]
[Means for Solving the Problem]In order to solve an aforementioned problem, the following composition is adopted in this invention.
(1) Are a low-temperature process of an alumina crystalline thin film, and a chrome oxide

Shimoji thin film is beforehand formed in polycrystal or a monocrystal substrate, or a base material in 800 ** from a room temperature by a sputtering technique, A low-temperature process of an alumina crystalline thin film forming an alumina (aluminum$_2O_3$) crystalline thin film by a sputtering technique on it in the temperature requirement concerned.
(2) A low-temperature process of an alumina crystalline thin film of the aforementioned (1) statement carrying out the weld slag of a chrome oxide target and the alumina (aluminum$_2O_3$) target into inactive gas.
(3) A low-temperature process of an alumina crystalline thin film of the aforementioned (1) statement carrying out reactive sputtering of a chromium (Cr) metal target and the aluminum (aluminum) metal target into discharge gas containing oxygen.
(4) The above (1), (2), or a low-temperature process of an alumina crystalline thin film given in (3) whose alumina crystalline thin film is alpha phase alumina membrane.
[0005]
[Embodiment of the Invention]Then, it explains still in detail about this invention. As mentioned above, this invention forms a chrome oxide crystalline thin film in a substrate or the base material by which a tunic is carried out beforehand, and relates to the method of manufacturing an alumina crystalline thin film at low temperature on it. What is done for the weld slag of this invention only with inactive gas using a compound target like chrome oxide and alumina, Or it is related with the method of manufacturing an alumina crystalline thin film at low temperature, by carrying out reactive sputtering into the inactive gas which contains oxygen using chromium (Cr) and an aluminum (aluminum) metal target. By an above-mentioned method, this invention is the most stable in an alumina crystal phase, and relates to the method of manufacturing most excellent alpha phase alumina membrane of the characteristic.
[0006]By the above-mentioned sputtering method, a chrome oxide (for example, Cr_2O_3) crystal phase thin film is formed in 800 ** from a room temperature. The Cr_2O_3 crystal as a Shimoji thin film, An alumina crystalline thin film and the effect that formation of alpha phase alumina membrane becomes easy especially are crystallographically acquired on it by having the same crystal structure as alpha alumina, and forming a chrome oxide Shimoji thin film in a substrate or a base material, since the difference of a lattice constant is only several percent. Since a chrome oxide crystal has only that mechanical properties, such as hardness and an antiwear characteristic, are similar with alumina, and thickness slight as a Shimoji thin film, it has an advantage which does not have big influence in the overall characteristic of an alumina crystalline thin film.
[0007]SUTOIKIO whose formed thin film it is very easy to operate it in the case of the method of carrying out RF weld slag only with inactive gas in the above-mentioned method using chrome oxide and an alumina compound target, and also almost corresponded with the target presentation -- it becomes metric chrome oxide and alumina. However, in RF weld slag using a compound target, it may be said that a membrane formation rate is a little low. In order to improve it more, the method of carrying out a reactivity direct current or RF weld slag is adopted into the inactive gas which uses chromium and an aluminum-metals target and contains oxygen. When it being necessary to optimize a process so that a Cr_2O_3 crystal and an alumina crystal can be formed, and controlling especially the ratio of oxygen and inactive gas by this method precisely manufactures a desired thin film material, it is the most important. In the method of this

invention, although the base material of polycrystal or a monocrystal substrate, and others is used, those kinds in particular are not restricted.
[0008]Although a general-purpose sputter device is used for the above-mentioned thin film production, It is possible to use it regardless of the kind, if the most important point among this inventions is a low-temperature process of the alumina crystalline thin film which used chrome oxide as the Shimoji thin film and is a device which can enforce the above-mentioned method, and it is not limited in particular for a device.
[0009]Although Cr_2O_3 of a crystalline form is used for the above-mentioned thin film production as a chrome oxide Shimoji thin film, for example, In addition, in [it is possible to use CrO, Cr_3O_4, CrO_2, and Cr_2O_5, CrO_3, etc., and] this invention, A chrome oxide Shimoji thin film may be what kind of chrome oxide compound, and the kind in particular is not limited. Although Cr_2O_3 is used for a ground compound target by the above-mentioned thin film production, for example, In addition, in [it is possible to use CrO, Cr_3O_4, CrO_2, and Cr_2O_5, CrO_3, etc. and] this invention, The ground compound target should just be a chrome oxide compound target, and the kind in particular is not limited. Although pure chromium (Cr) metal is used for a metal target by the above-mentioned thin film production, for example, It may be possible to use other metal (for example, aluminum) and the alloyed thing, a chrome metal target may be what kind of alloy target which uses not only a pure chrome metal but chromium as the main ingredients in this invention, and the kind in particular is not limited.
[0010]The low-temperature process of the alumina crystalline thin film by a described method has the following features.
(1) Formation of alpha phase alumina membrane is accepted from about 200 ** near substrate temperature.
(2) alpha phase alumina membrane with a precise structure is formed.
[0011]
[Example]Then, this invention is concretely explained based on an example.
Generalized magnetron sputtering equipment was used for thin film production in example 1(1) method this example. Even three cathodes can arrange to the device concerned and power controls can be arbitrarily done for it in an RF generator or DC power supply at each. A substrate can be rotated and substrate temperature is precisely set up from a room temperature to 800 **. The chrome oxide target (Cr_2O_3, phi50mm, 99.9% of purity) of one set of marketing of a cathode and the alumina target (aluminum$_2O_3$, phi50mm, 99.99% of purity) of one more set of marketing were installed. After exhausting a vacuum system below to $2.5x10^{-6}$Pa, only argon gas was introduced and membrane formation was performed after 30-minute pre weld slag with the total pressure of 0.1 Pa. Substrate temperature was set as the range from a room temperature to 800 **, and glass, a silicon single crystal, sapphire, etc. were used as a substrate.
[0012]That is, the high-frequency power 150W was first applied to the chrome oxide target, and the Cr_2O_3 thin film about 200 nm thick was formed on the substrate. The chrome oxide thin film formed at each temperature by the X-ray diffraction method was analyzed.
[0013]After coating beforehand about 200 nm of chrome oxide crystalline thin films, the high-frequency power of 150W was applied as well as the alumina target, weld slag was performed, and alumina membrane was formed about 300 nm in thickness. The crystal phase was identified for the thin film with the formed multilayer structure with the X-ray

diffraction method.
[0014](2) The X diffraction pattern of the chrome oxide thin film formed on the silicon substrate by the method of the result above is shown in drawing 1. Formation of the Cr_2O_3 crystalline thin film was checked from a room temperature to 600 **. The X diffraction pattern of the chrome oxide / alumina multilayered film created on the silicon substrate by the above-mentioned method is shown in drawing 2. It is clear from the result which diffraction peaks other than chrome oxide were seen from about 200 **, and was compared with a standard [for JCPDS] one that output's it is an alpha alumina thin film so that it may turn out that it compares with drawing 1. The same result was obtained, as a result of carrying out reactive sputtering of a chrome metal target and the aluminum-metals target into the inactive gas containing oxygen and analyzing them similarly.
[0015]In the comparative example 1 above-mentioned example 1, without using a chrome oxide Shimoji thin film, the high-frequency power of 150W was applied as well as the alumina target, and alumina membrane was directly formed about 300 nm in thickness on the silicon substrate. The X diffraction pattern of the alumina membrane formed at each temperature from a room temperature to 700 ** is shown in drawing 3. A diffraction peak with clear all was not seen and formation of the alumina crystal phase was not seen to 700 **.
[0016]as mentioned above -- unless the composition which this invention is not limited to said example carried out, and was indicated to the claim although this invention was explained based on the example is changed -- how -- also coming out -- it can carry out.
[0017]
[Effect of the Invention]As explained in full detail above, this invention carries out the tunic of the chrome oxide which can form a crystal phase from a room temperature, and has a crystal structure very similar to an alumina crystal on a substrate beforehand, and relates to the new low-temperature process of the alumina crystalline thin film which forms alumina membrane on it.
By the bottom of the low temperature of 2 forming temperature which can form an alumina crystalline thin film and alpha phase alumina membrane which was the most stable and was especially excellent in the characteristic at low temperature by this invention farther than the method of 1 former. 3 in which the kind of the substrate which can be chosen, or base material spreads greatly -- the effect that the application to the industrial world, such as providing the abrasion proof in machinery industry, semiconductor industry, etc., the alumina hard coating aiming at protection, or the alumina substrate of the quality of high cheaply, is expected greatly by that cause is done so.

[Translation done.]